

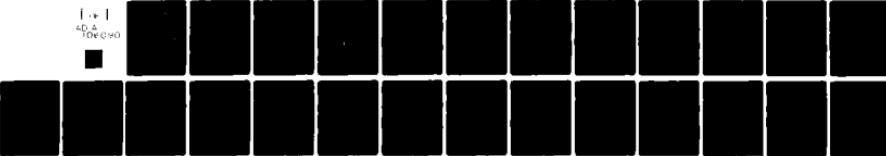
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MASS SPECTROMETRIC STUDIES OF THE THERMAL DECOMPOSITION OF SOME AZIDO POLYMERIC BINDERS

Milton Farber and R. D. Srivastava

ANNUAL SUMMARY REPORT

Department of the Navy
Office of Naval Research
Arlington, Virginia 22217

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September 1981

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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Thermal decomposition studies were conducted on BAMO, GAP and AZOX. Decomposition was initiated at approximately 120 C, with activation energies for the three materials approximately 170 kJ/mol ¹ . The primary mechanism for the decomposition is the release of molecular N ₂ . Secondary decomposition occurring at temperatures above 150 C included the fracturing of the three-carbon backbone of both the polymers and the monomer. Higher decomposition temperature species included HCN, HCO, and smaller amu fragments. | | |

1. INTRODUCTION

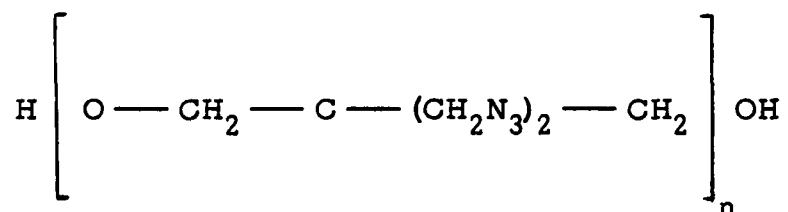
During the past twelve months thermal decomposition studies were performed on several energetic azide monomers and polymers. These included 3,3,bis(azidomethyl) oxetane polymer (BAMO), glycidyl azide polymer (GAP), and azido oxetane monomer (AZOX). The materials were synthesized at various laboratories, which supplied samples: SRI International (BAMO), Edwards Air Force Base (GAP), and Fluorochem (AZOX).

The monomers and polymers all begin to decompose with the release of molecular nitrogen at approximately 120 C. Their activation energies for the primary mechanism of thermal decomposition, the breaking of the azido bond, were in the neighborhood of 170 kJ mol^{-1} (40 kcal/mole).

2. THERMAL DECOMPOSITION STUDIES ON AZIDO MONOMERS AND POLYMERS

2.1 Thermal Decomposition of BAMO-BDO, 3.2K

The thermal decomposition of BAMO,



was studied by means of weight loss and mass spectrometry. The weight loss experiments involved placement of a capsule within an alumina effusion cell, as shown in Fig. 1. The cell was maintained at a constant temperature and products were released through the orifice into the vacuum chamber via an elongated orifice for molecular beam formation.

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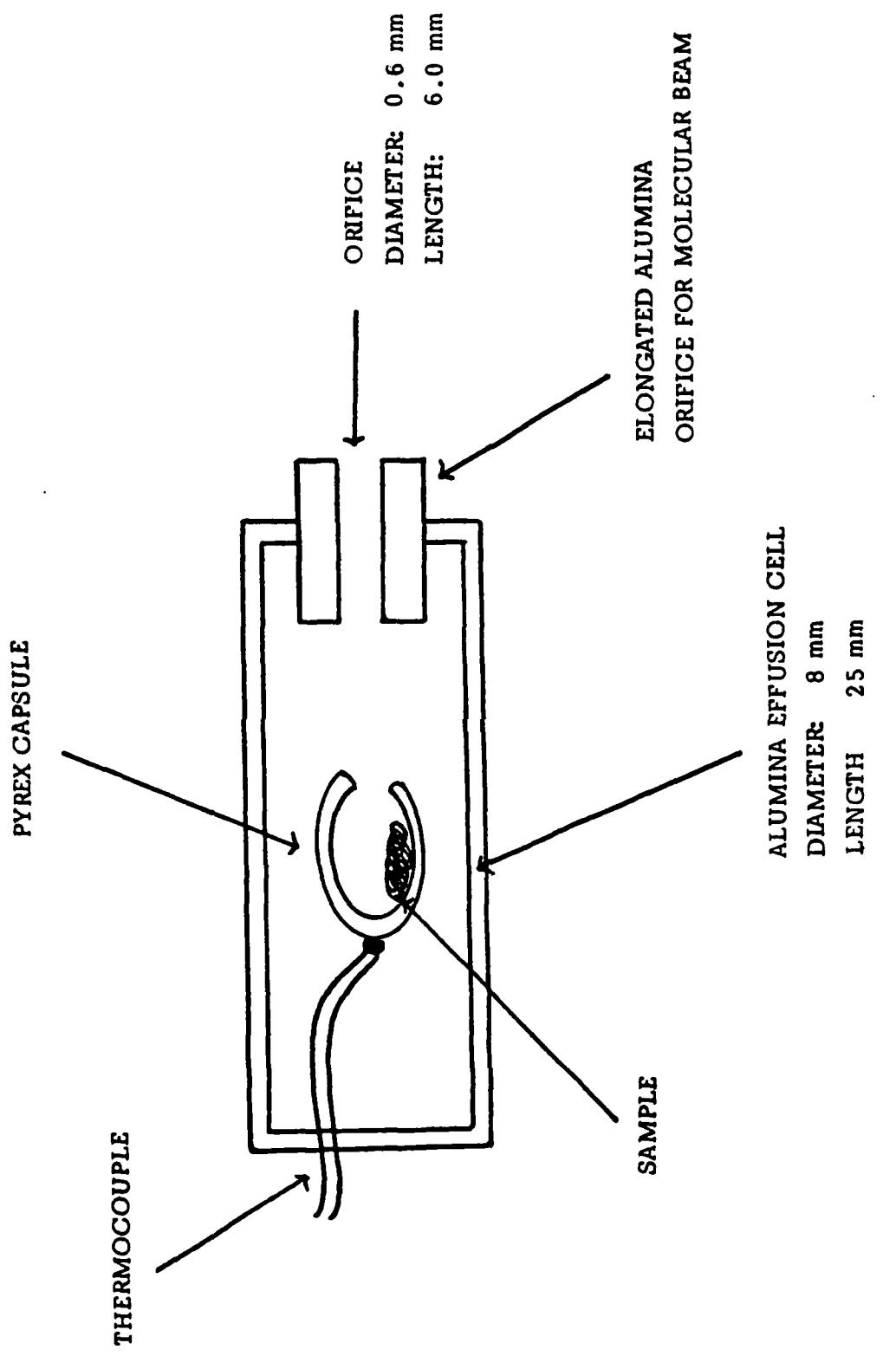


Fig. 1. Sample capsule within effusion cell with elongated orifice

Weight loss experiments on 25 mg samples of BAMO were performed for periods of 3 hours each at temperatures of 82, 100, 118, 130, 135, and 202 C. These data are presented in Table 1. No apparent decomposition occurred at 80, 100 or 120 C, although at 120 C the sample turned a slightly off-white, or cream, color from its original white. Slight decomposition began at 130 C, with nearly complete decomposition at 200 C, where the rate of decomposition was 20 times that at 130 C. A 25 mg sample maintained at 200 C for three hours lost 80% of its initial mass, leaving behind a carbonaceous tar residue. The melting point of the BAMO-BDO was found to be 75 C.

The release of molecular nitrogen can be seen as low as 130 C, and has a good temperature dependence above 160 C, depicted in Fig. 2., which shows the decomposition as a function of temperature quite clearly. At any given temperature the decomposition rate as a function of temperature is essentially constant, whether in a heating or cooling cycle. Figure 3 shows the decomposition of a 25 mg sample maintained at a temperature of 202 C over a two-hour period. As can be seen, for the first 1-1/2 hours the rate of N_2 evolution remains nearly constant, but as the sample becomes depleted during the last 30 minutes the N_2 concentration decays to a negligible amount.

An activation energy study of the primary decomposition path, the release of N_2 from the azide groups, was also completed. Figure 4 depicts the N_2 concentration as a function of temperature, while Fig. 5 is a plot of the N_2 intensity versus the reciprocal of the absolute temperature. The E_a for the BAMO-BDO, 3.2K, was determined as $178.7 \text{ kJ mol}^{-1}$ (42.7 kcal/mole). From this study the primary mechanism for BAMO decomposition

Table 1
Thermal Decomposition of BAMO-BDO

| Temp. (°C) | Experiment Duration (hours) | Initial Sample Weight (gms) | Final Sample Weight (gms) | Rate of Decomposition (g/sec) | Appearance |
|---------------|-----------------------------------|-----------------------------------|---------------------------------|-------------------------------------|-----------------------------------|
| 82 | 3 | 0.0226 | 0.0226 | 0 | No color change; sample melted |
| 100 | 3 | 0.0226 | 0.0226 | 0 | " " |
| 118 | 3 | 0.0226 | 0.0226 | 0 | Slightly off- white |
| 130 | 3 | 0.0248 | 0.0235 | 1.2×10^{-7} | Cream colored |
| 135 | 3 | 0.0347 | 0.0309 | 3.5×10^{-7} | " " |
| 202 | 3 | 0.0252 | 0.0045 | 19.1×10^{-7} | Brownish-black char |

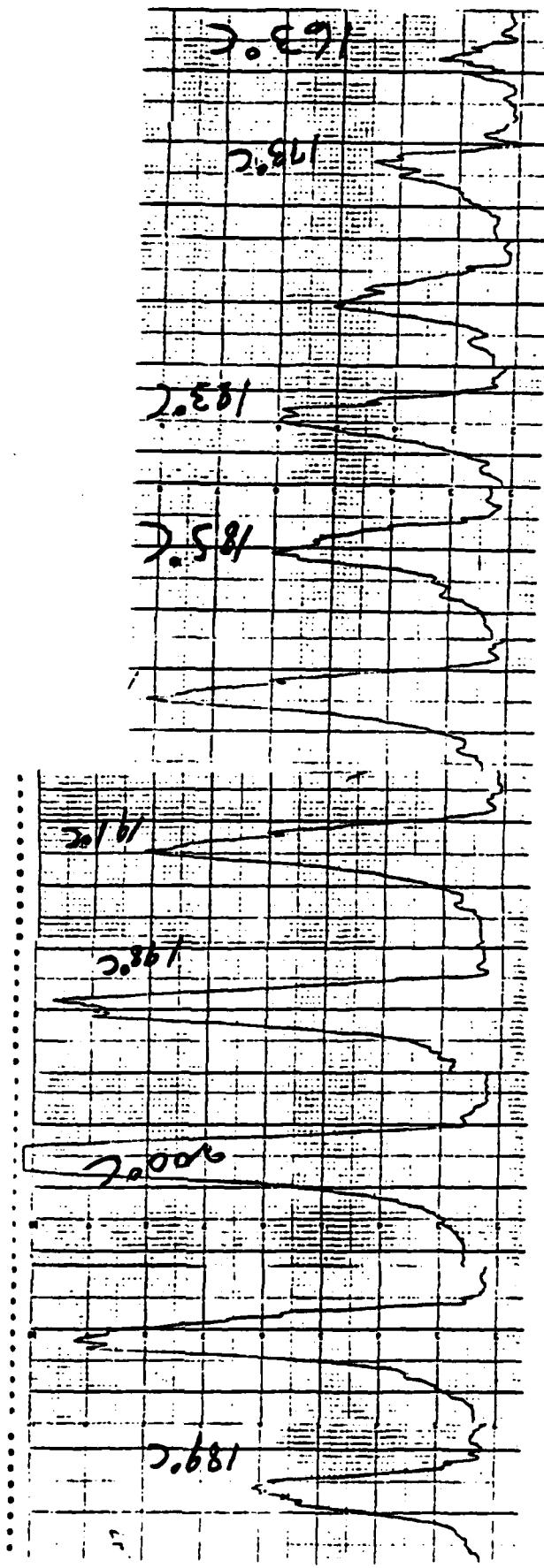


Fig. 2. Thermal Decomposition of BAMO-BDO as a Function of Temperature

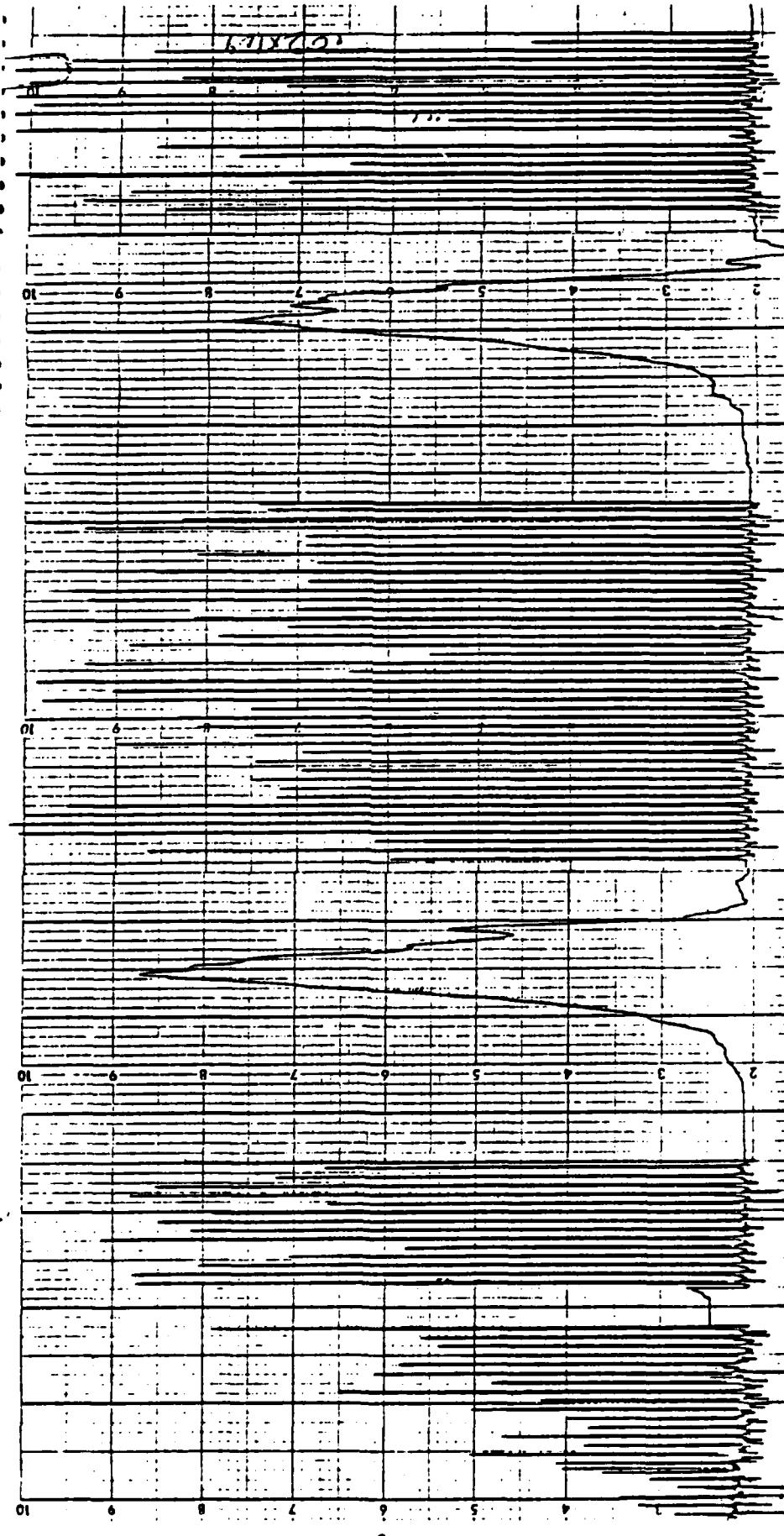


Fig. 3. Thermal Decomposition of BAMO-BDO. A 25 mg sample was heated at a constant temperature of 202°C for 2 hours. For the first 1-1/2 hours the rate of N₂ evolution was nearly constant (each line represents a time span of 1 minute). During the final 30 minutes the N₂ evolution showed a steady decline as the sample became depleted.

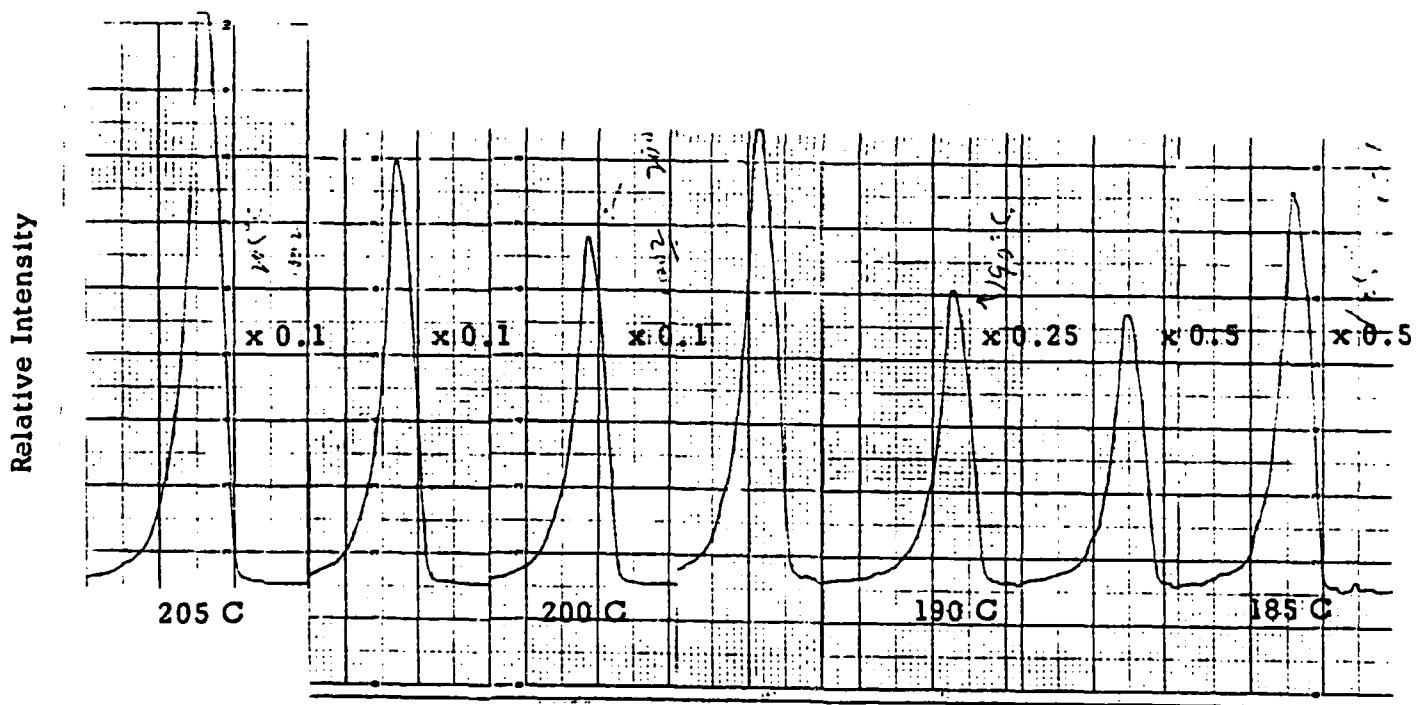
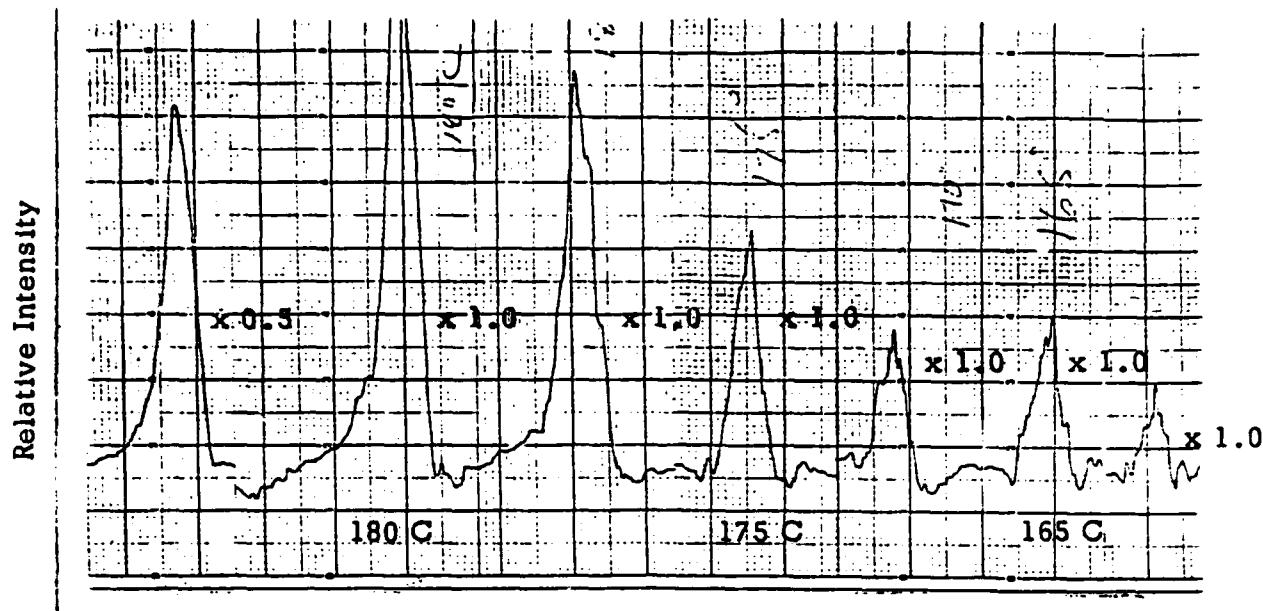


Fig. 4. Decomposition of BAMO-BDO as a Function of Temperature.

The peaks represent the release of molecular nitrogen.

(These graphs are a composite of the individual peak heights
and are not continuous as a function of temperature.)

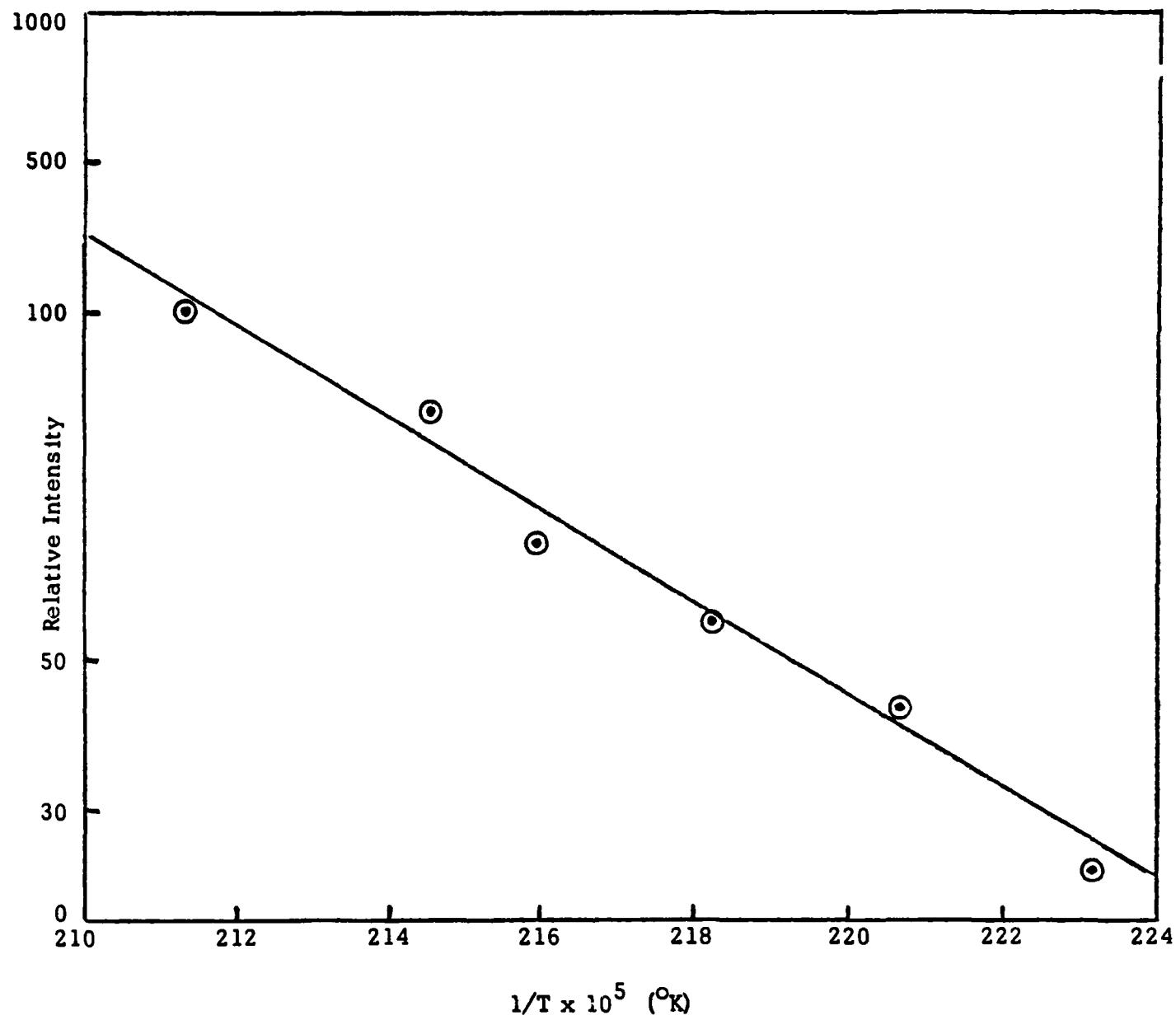
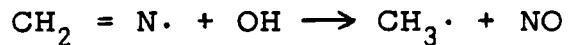


Fig. 5. Relative Intensity of N_2 from the Thermal Decomposition
of BAMO-BDO plotted as a Function of the Reciprocal
of the Absolute Temperature ($1/T^{\circ}\text{K}$)

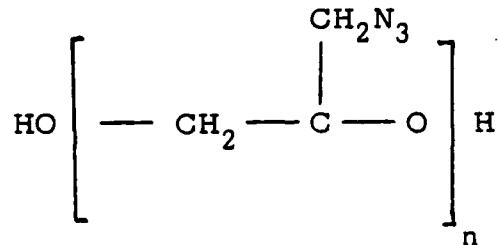
is the release of N_2 from the rupture of the azido bond. The three-carbon backbone of the polymer appears to remain intact initially since other gaseous species are not observed until the sample is heated to higher temperatures.

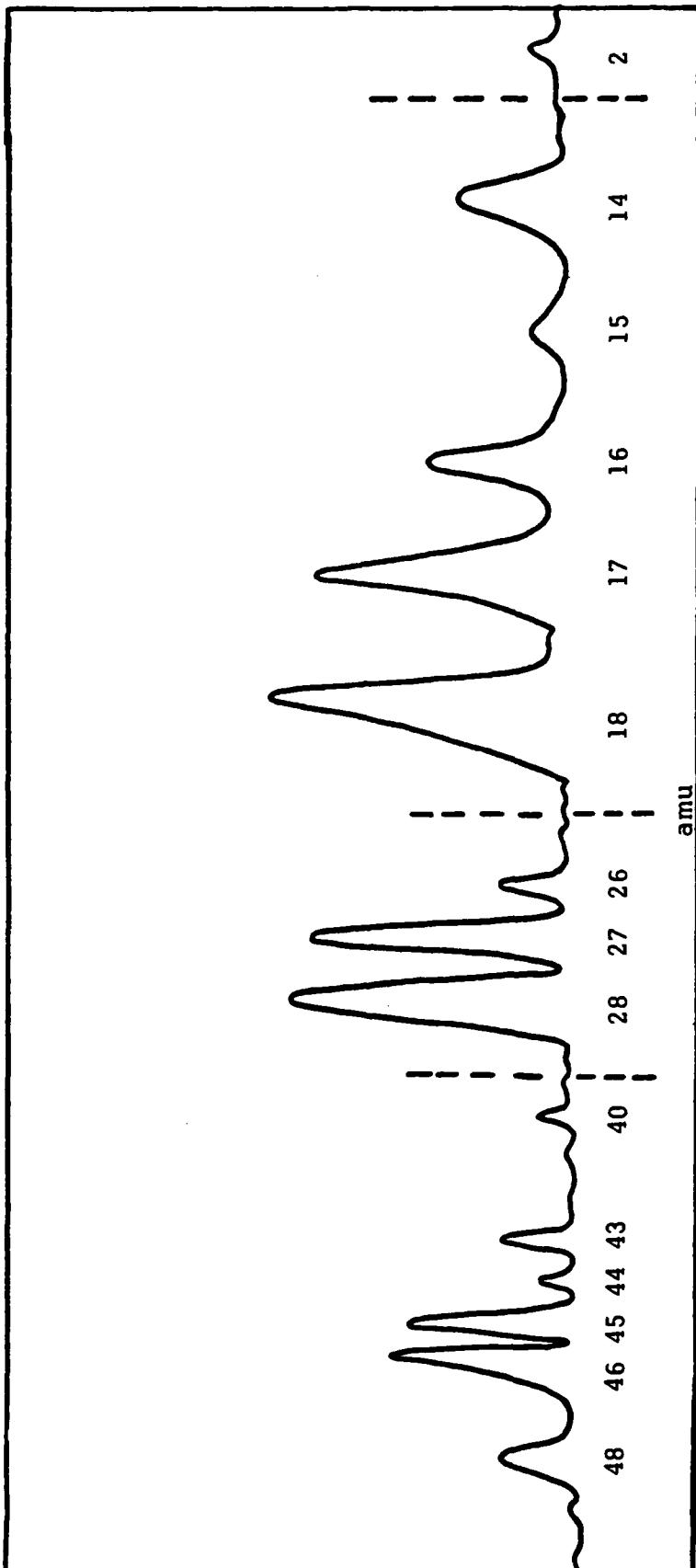
In addition to molecular N_2 , the mass spectrometer showed peaks corresponding to HCN as well as peaks of amu 2, 14, 15, 16, 17, and 18 which may be attributed to H_2 , CH_2 , CH_3 , O (fragment of O_2), OH and H_2O , respectively. Some low intensity peaks in the amu 40 to 46 range were also observed, indicating thermal decomposition of the hydrocarbon backbone. The temperature range of this secondary decomposition was 200 C. The intensities in Fig. 6 are only relative within the dashed lines. The intensity peaks in the 40 to 48 amu range are effusion cell reactions resulting in H, C, O, and N variations. The formation of NO_2 , for example, may require cell reactions of the following type:



2.2 Thermal Decomposition of GAP

The initial investigation of the thermal decomposition of glycidyl azide polymer, GAP





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Fig. 6. PRODUCTS FROM THE THERMAL DECOMPOSITION OF BAMO
PEAK HEIGHTS ARE ONLY RELATIVE WITHIN DESIGNATED SECTIONS.

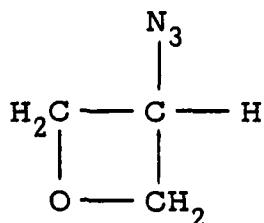
was a study of its primary decomposition as a function of temperature.

As in the case of BAMO, the GAP appears to be fairly stable to 120 C, at which molecular N₂ is released.

The results of the decomposition study of this polymer as a function of temperature is shown in Fig. 7. For a 20 C rise in temperature the rate of N₂ evolution is increased by approximately a factor of 10. In the amu range 24 to 32 the major ion intensity constituent is molecular N₂. This appears to be the same pattern as found in the thermal decomposition of BAMO. However, at temperatures above 170 C a slight intensity of amu 27, HCN, is observed, approximately 1 to 2% of the N₂ intensity. It is likely that secondary decomposition begins at a slightly lower temperature for GAP than for BAMO. The primary decomposition mechanism for GAP is N₂ release. A plot of the log N₂ against 1/T, as shown in Fig. 8, yields an activation energy of 176.6 kJ mol⁻¹ (42.2 kcal/mole), nearly identical to the E_a of BAMO.

2.3 Thermal Decomposition of AZOX Monomer

The thermal decomposition of azido oxetane monomer, AZOX



as a function of temperature was investigated by the double boiler-effusion technique. The AZOX was maintained in an ice bath and the vapor at 0 C was allowed to enter the effusion cell where it was heated to a

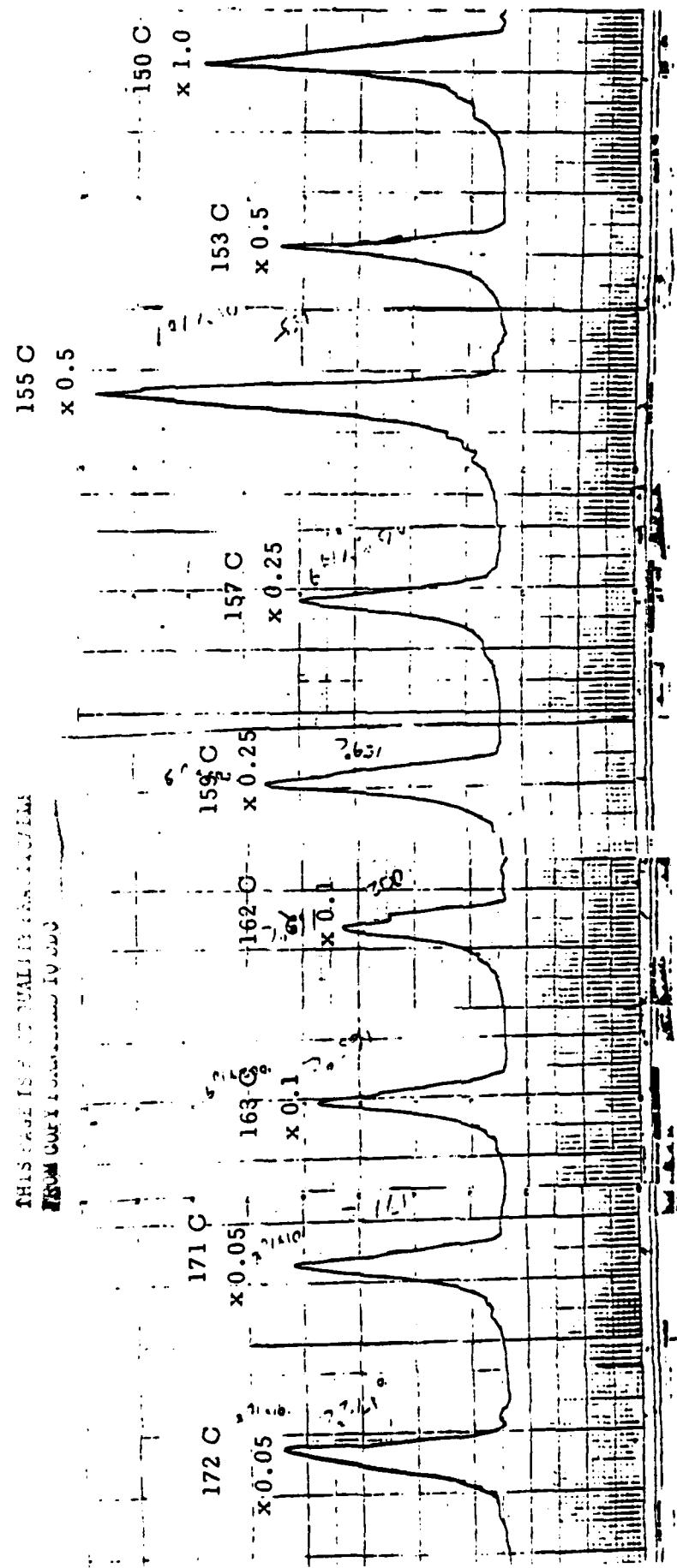


Fig. 7. Decomposition of GAP as a function of temperature.

The amu range is 24 to 32. The central peaks are molecular nitrogen. These graphs are composites of the individual peak heights and are not continuous as a function of temperature.

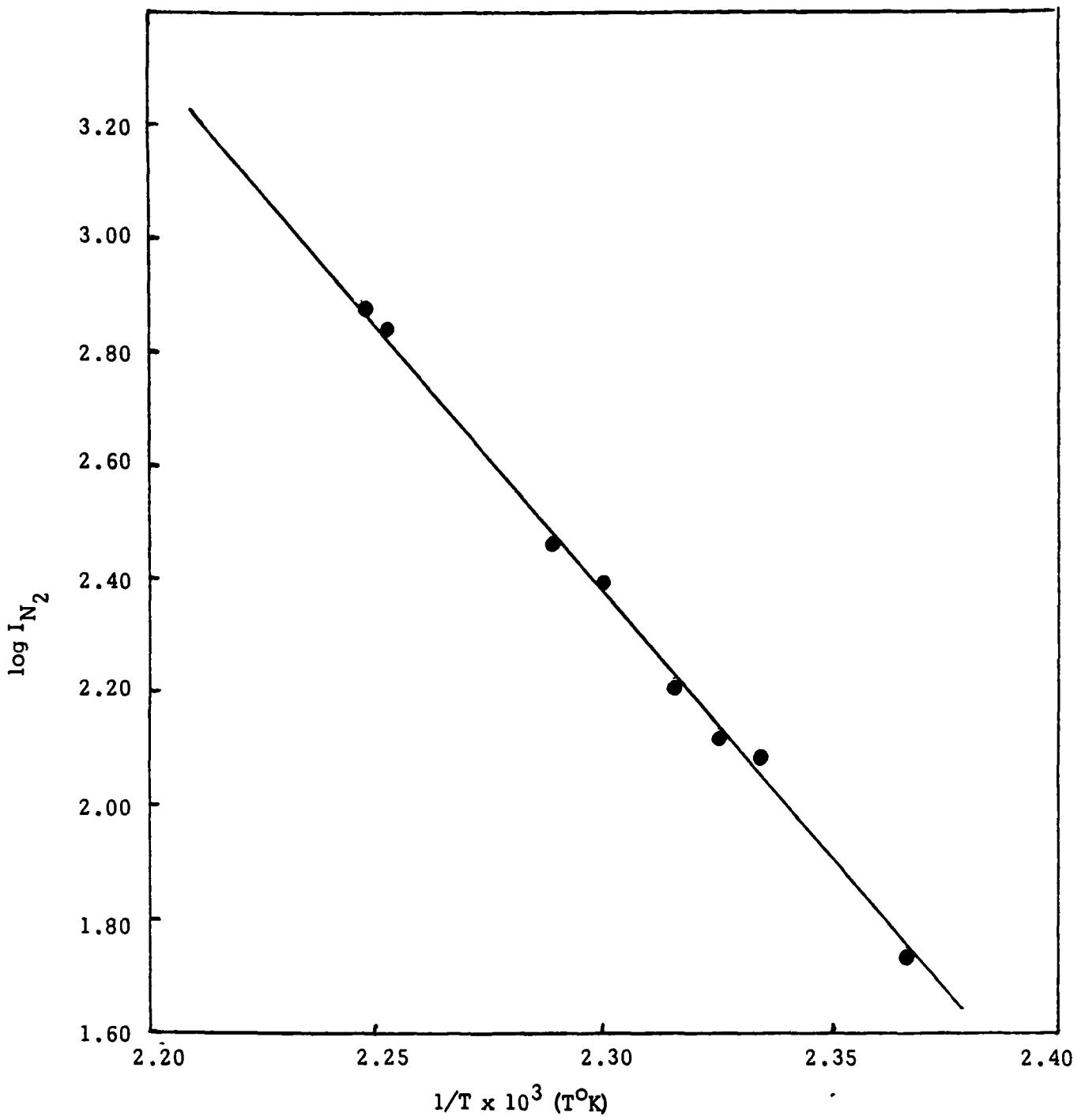


Fig. 8. Log of the relative intensity of N_2 from the thermal decomposition of glycidyl azide polymer (GAP) plotted as a function of the reciprocal of the absolute temperature ($1/T^\circ K$). Activation energy = $176.6 \text{ kJ mol}^{-1}$ (42.2 kcal/mole).

given temperature. The molecular beam of the decomposition products was introduced into the mass spectrometer and relative intensity measurements were made. Prior to heating the AZOX a mass spectrum showed a major peak occurring at 71 amu, which is the AZOX monomer backbone after release of N_2 due to electron impact dissociation within the ionization chamber of the mass spectrometer.

The monomer vapor was heated and the 24 to 32 amu range was monitored with the mass spectrometer. As can be seen in Fig. 9, the major species was N_2 , as it was in the BAMO and GAP studies. However, at temperatures above 160 C two small but prominent peaks appeared at amu 27 and 29, apparently HCN and HCO. This indicates that although the release of N_2 from the azido group is the primary decomposition mechanism, the three-carbon backbone loses stability when the azide bonds are broken. The major difference between this molecule and the BAMO and GAP moieties is that the azide groups are attached to the backbone whereas in the other two they are attached to methyl groups outside the three-carbon structure. These results agree with the findings of Dr. M. B. Frankel and colleagues.¹

A plot of the log of N_2 intensity against the reciprocal of the absolute temperature (Fig. 10) yielded an activation energy of $167.8 \text{ kJ mol}^{-1}$ (40.1 kcal/mole). This apparently is indicative of azide monomers, as shown in a recent publication by Isayev, et al.² They reported E_a values of 39 kcal/mole for several aliphatic azides including β -triazoethanol, 1,3,diazide propanol, 1,3,diazide propylene ester of acetic acid, and 1,3,diazide propylene acid.

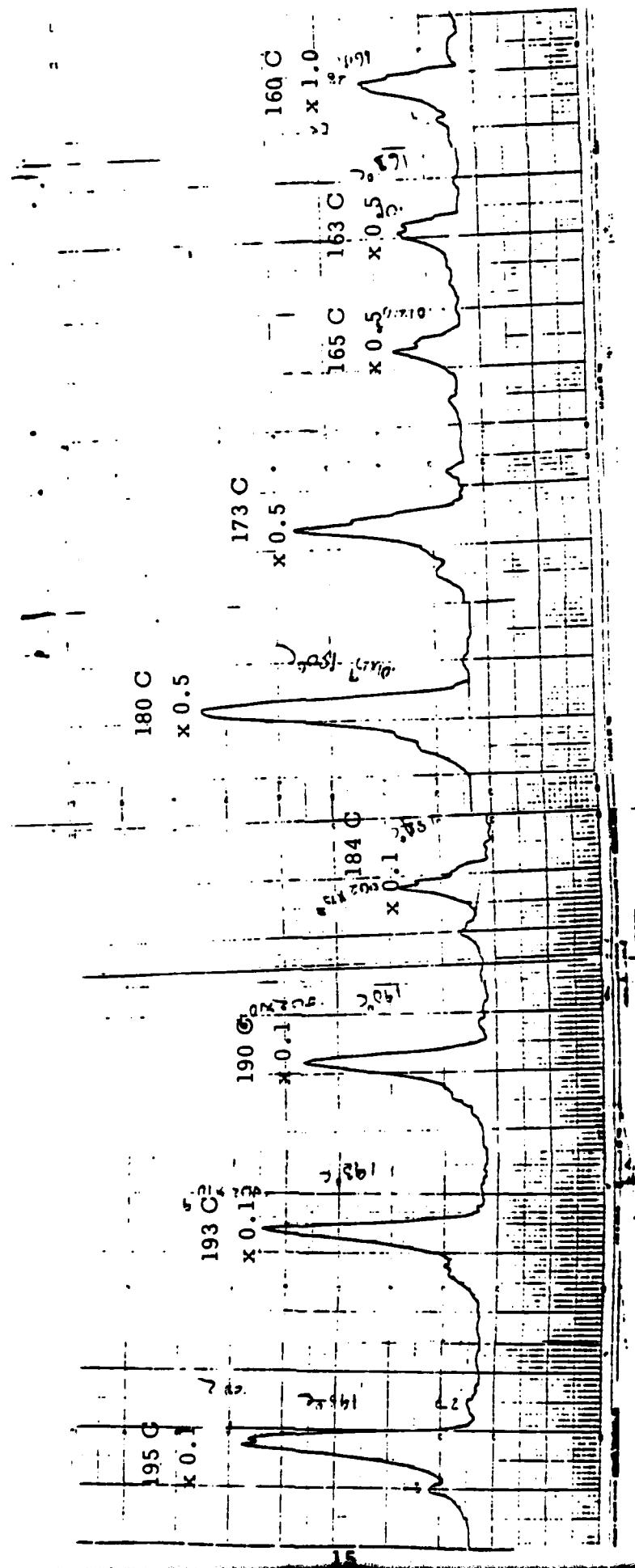


Fig. 9. Decomposition of AZOX monomer as a function of temperature.
 The amu range is 24 to 32. The central peaks are molecular nitrogen, with smaller peaks on each side of HCN at 27 amu and HCO at 29 amu. These graphs are composites of the individual peak heights and are not continuous as a function of temperature.

Mass Spectra
24-32 amu
27 amu
29 amu

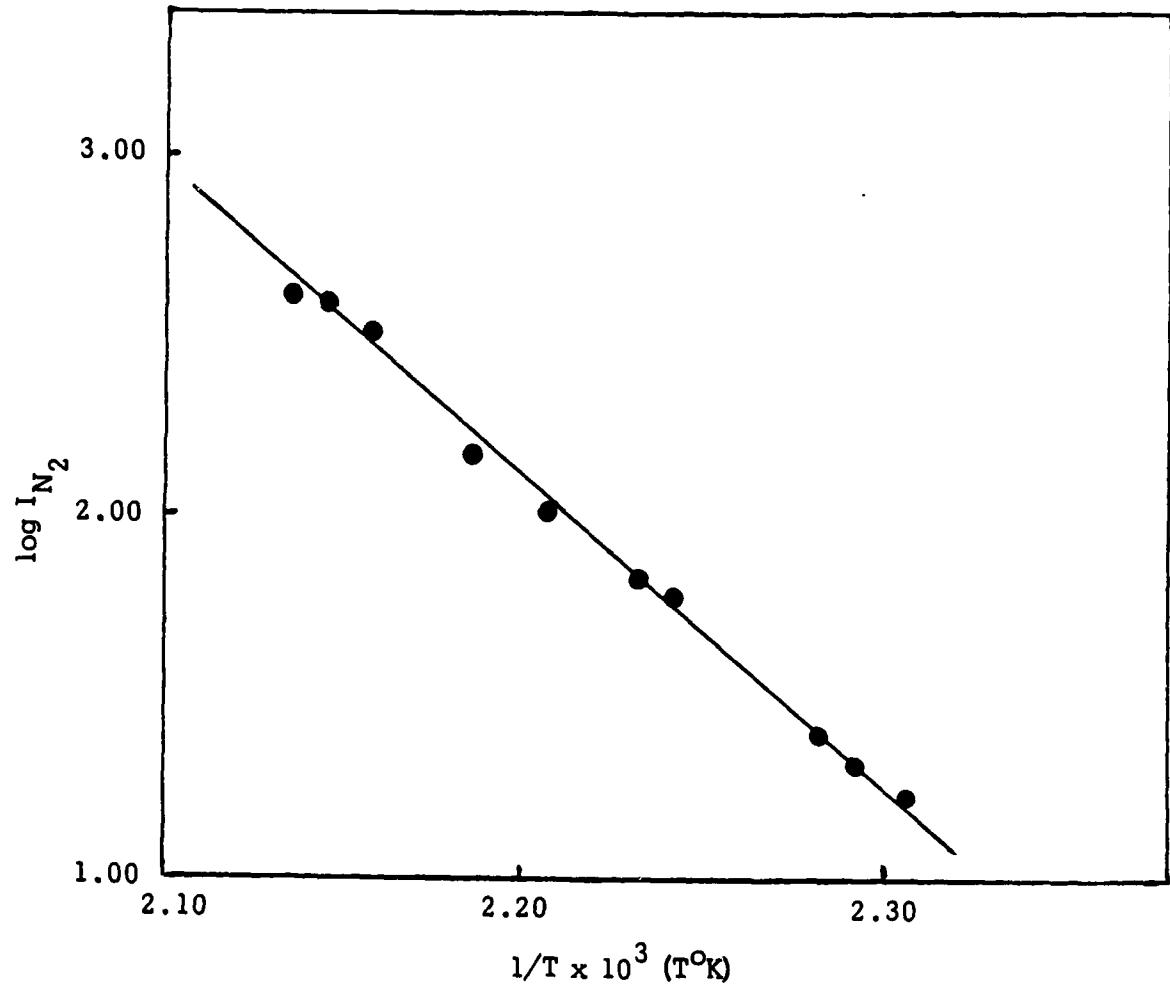


Fig. 10. Log of the relative intensity of N_2 from the thermal decomposition of azido oxetane monomer (AZOX) plotted as a function of the reciprocal of the absolute temperature ($1/T^{\circ}\text{K}$). Activation energy = $167.8 \text{ kJ mol}^{-1}$ (40.1 kcal/mole)

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